GEORGIA INSTITUTE OF TECHNOLOGY — OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[X] ORIGINAL [ ] REVISION NO. _____

Project No. A-3966 GTRI/~~GIT~~ DATE 9/25/84

Project Director: Dr. T.L. Thomas ~~Sch.of~~Lab. EMSL

Sponsor: Wil Gas Company

Type Agreement: Standard Industrial Agreement dated 8/24/84

Award Period: From 9/11/84 To 10/3/84 (Performance) 10/3/84 (Reports)

Sponsor Amount:	This Change	Total to Date
Estimated:	$ 1,610	$ 1,610
Funded:	$ 1,610	$ 1,610

Cost Sharing Amount: $ none Cost Sharing No: N/A

Title: Evaluation of Zeolite Samples

ADMINISTRATIVE DATA OCA Contact Brian J. Lindberg x4820

1) Sponsor Technical Contact:

Mr. Jerry Callens
District Manager
Wil Gas Company
Route 4, Box 75B
La Grange, Texas 78945

2) Sponsor Admin/Contractual Matters:

same as 1)

Defense Priority Rating: N/A Military Security Classification: N/A

(or) Company/Industrial Proprietary: N/A

RESTRICTIONS

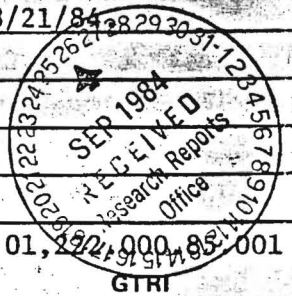
See Attached N/A Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval — Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with none proposed or anticipated.

COMMENTS:

100% advance payment received by check No. 900167 dated 8/21/84



I.D. No. 01,290,000,85,001

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GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

Date 4/13/85

Project No. A-3966 ~~School~~/Lab EMSL

Includes Subproject No.(s)

Project Director(s) Dr. T. L. Thomas GTRI / ~~GTRI~~

Sponsor Wil Gas Company

Title Evaluation of Zeolite Samples

Effective Completion Date: 10/3/84 (Performance) 10/3/84 (Reports)

Grant/Contract Closeout Actions Remaining:

- [ ] None
- [X] Final Invoice or Final Fiscal Report
- [ ] Closing Documents
- [ ] Final Report of Inventions
- [ ] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other

Continues Project No. Continued by Project No.

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Form OCA 60:1028

A-3966

Georgia Institute of Technology
ENGINEERING EXPERIMENT STATION
Atlanta, Georgia 30332

4 October 1984

Mr. Jerry Callens, District Manager
Wil Gas Co.
Route 4, Box 75-B
La Grange, Texas 78945

A-3966/DR. THOMAS

Dear Jerry:

As discussed with you by telephone, we have completed the laboratory work under our Research Proposal No. ME-OD-2386, which involved the zeolite samples you sent to us covered by your letter to me dated July 27, 1984.

After discussion with you, we elected to evaluate samples taken from the top and bottom of your B-bed (labeled "B-top) and "B-bottom") and for comparison, in identical tests, evaluated your original commercial molecular sieve put into this B adsorber (labeled by you "Unused") and another commercial molecular sieve (Union Carabide Type 4A, 1/8-inch pellets). The results of these tests are shown in the attached Table I.

The significant facts derived from our laboratory evaluation can be summarized as follows:

1. You indicated that the "Unused" sample was part of the original commercial molecular sieve that was charged to your dehydrators but had spilled onto ground and was recovered when you sent us the sample. On activation of this "Unused" at 350°C under vacuum prior to testing for water and oxygen adsorption, this material turned gray and released organic vapor which deposited on the cooler walls of the vacuum system.

2. Both "B-top" and "B-bottom" contained coke with more coke on the bottom sample where your hot regeneration gas entered.

3. Water equilibrium capacity of these coked materials was reduced by about 50% but recovered most of this lost capacity after burn-off of the coke.

4. Reduction in oxygen equilibrium adsorption capacity is a measure of pore closure which can occur with Type 4A in the presence of water vapor at higher temperatures. Such pore closure does not affect water equilibrium capacity but can significantly reduce the rate at which water is adsorbed. As seen in Table I, oxygen adsorption capacity for top and bottom samples was severely depressed and did not recover on burn-off.

5. Crystallinity as measured from the X-ray patterns appears normal except for "Unused" sample, for which no conclusion can be drawn because of its unknown history and its evolution of organic material on heating (see item 1 above).

6. Ratio of the intensities of the 110 to 100 peaks in the X-ray pattern is another means of determining pore closure. The reversal of these peaks in the B-top and B-bottom samples (ratio of 1.05) from fresh Taype 4A (ratio of 0.69) is the result of pore closure.

The following conclusions can be made from the discussion and technical facts presented above:

1. The "Unused" material has low X-ray crystallinity and evolved organic vapors on heating. If these facts are the result of what happened to this material between the time it was spilled on loading of the adsorbers and the time you sent it to us, these data can be disregarded; however, of these facts are characteristic of the original material as purchased by you, we must conclude it is unacceptable product for natural gas treating.

2. All molecular sieve adsorbent will coke to some extent when used in service similar to yours. The 9.1 wt-% does, however, seem high. This coke has reduced the water equilibrium capacity of your beds but is not the sole cause of your problem.

3. The second cause for your beds to loss effectiveness is that the rate of adsorption of water is reduced by pore closure of the Type 4A in your adsorbent. This contributes to lengthening of the mass transfer zone in your leads and beds to premature break-thru. Good commercial Type 4A products should not pore close, under the operating conditions you report for your dehydrators.

Sincerely,

T. L. Thomas, Head
Zeolite Research Program

TLT: ae

TABLE I

	Unused	B-bottom	B-top	UCC
weight-% coke	--	9.1	3.3	--
Water capacity (17.5 mm Hg, 25°C) as weight-% adsorbed				
-as received	25.0	10.6	13.5	24.2
-burned off	--	23.9	19.7	--
Oxygen capacity (75 torr, -196°C) as weight-% adsorbed				
-as received	26.6	4.3	4.7	24.9
-burned off	--	8.0	5.4	--
crystallinity (X-ray) as % of Type A powder				
-as received	47.2*	73.6	81.0	76.6
-burned off	--	80.0	78.1	--
ratio I_{110} to I_{100} lines in X-ray pattern				
-as received	0.46*	1.05	1.07	0.69
- burned off	--	0.72	0.64	--

*questionable since history prior to being received is unknown.